

**COMMERCIAL-SCALE DEMONSTRATION OF THE  
LIQUID PHASE METHANOL (LPMEOH™) PROCESS**

**TECHNICAL PROGRESS REPORT NO. 24**

**For The Period**

**1 April - 30 June 2000**

**Prepared by**

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Allentown, Pennsylvania**

**and**

**Eastman Chemical Company  
Kingsport, Tennessee**

**for the**

**Air Products Liquid Phase Conversion Company, L.P.**

**Prepared for the United States Department of Energy  
National Energy Technology Laboratory  
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## **Abstract**

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman chemicals-from-coal complex in Kingsport.

The LPMEOH™ Demonstration Unit operated at 95% availability throughout the quarter. A 2-hour forced outage was caused by the failure of the control valve which regulates the flow of boiler feedwater to the steam drum on the LPMEOH™ Reactor. An additional 77-hour forced outage was initiated by a leak on instrument tubing associated with the measurement of boiler feedwater flow to the steam drum. A power transient was also experienced which interrupted the operation of the LPMEOH™ Demonstration Unit and adjoining process units within the chemicals-from-coal complex.

A major catalyst withdrawal and addition campaign was undertaken during April and May of 2000 to raise catalyst activity. After the campaign, the total catalyst inventory was calculated to be 45,207 pounds.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average of 703 KSCFH. During these operating periods, the reactor pressure was set at between 700 and 710 psig and temperature was maintained at 235°C.

Three intervals of carbon monoxide (CO)-rich testing were also performed. A 24-day test was concluded on 13 April 2000 during which a syngas stream which contains primarily CO (CO Gas) was introduced with the Balanced Gas to achieve a ratio of hydrogen (H<sub>2</sub>) to CO in the reactor inlet of 1:1. On 09-10 May 2000, a 22-hour test was performed at a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.5:1. Reactor temperature was raised to 250°C during this test period to allow for greater catalyst productivity and to lower the purge rate. On 22 June 2000, a 6-hour test was conducted at a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.6:1.

There were two extended periods of operation at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 1.24% per day was calculated for the period 24 March to 12 April 2000, during which the reactor inlet gas with a H<sub>2</sub>/CO ratio of 1:1 was fed to the LPMEOH™ Reactor. An overall deactivation rate of 0.89% per day was calculated for the period 13 May and 10 June 2000, during which Balanced Gas alone was used as feed to the reactor. The results of these two data sets are statistically similar, given the scatter in the calculated values for the catalyst rate constant. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (AFDU) in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples; however, the size increase has stabilized or decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison), although low (less than 200 ppmw), had been increasing, but has now stabilized in the most recent samples.

Eastman has accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon to replace the manganese oxide which is currently used in the 29C-40 catalyst guard bed within the LPMEOH™ Demonstration Unit. This material was recommended by vendors as having affinity for arsenic, and this class of adsorbent should also be able to remove sulfur and metal carbonyl species from the syngas. Laboratory testing has confirmed the affinity of this carbon for arsenic; the capacity cannot be determined, as no on-line analytical technique exists to measure arsenic in a syngas stream to the parts-per-billion concentration. The changeout of the 29C-40 catalyst guard bed is scheduled for the week of 31 July 2000.

A test of the conditions for the proposed in-situ activation of catalyst in the LPMEOH™ Reactor was performed. The corresponding reactor inlet flowrate during the in-situ activation procedure is about 20% less than the basis which was used during confirmation of the procedure in the autoclave. These results will be evaluated to determine if additional laboratory or field testing will be required.

A test of the ability to ramp the LPMEOH™ Reactor from a standby condition to production rates was performed. This test attempted to simulate the ramping that must be performed within the startup requirements of an IGCC facility. A ramping rate of 3% of the design flow (990 KSCFH) per minute was achieved, which approaches the minimum target rate of 5% per minute. Based upon the experience gained from this test, it is expected that the goal of 5 to 10% change in design flow per minute, which meets the requirements for the IGCC system, can be met.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, has met the expectations for pressure drop and reactor operation.

During the reporting period, a total of 5,382,395 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 58.9 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on one of the project sites selected for the off-site, product-use test program. Testing continues on the

impact of the trace mineral oil in the stabilized methanol on performance of the catalyst in the reformer test apparatus at the University of Florida. A sample of fresh mineral oil was sent to the University; this material will be blended with chemical-grade methanol for testing in the reformer, and will be used to evaluate potential designs for an oil filtration and removal system which could be incorporated into the flowsheet for a phosphoric acid fuel cell system.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report is under development which presents the results of the Fall 1999 design verification test at the LaPorte AFDU.

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary in order to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. Work was initiated to update the Demonstration Test Plan, given the change in the term of the Cooperative Agreement.

Two DOE quarterly review meetings were held during the reporting period. Major topics included a review of the LPMEOH™ Demonstration Unit performance, an update on the status of the request for the no-cost time extension, and the results of the in-situ activation test and the ramping study.

A draft of the paper entitled “Catalyst and Process Development for Liquid Phase DME Synthesis” was submitted to DOE for review. This paper will be presented at 17<sup>th</sup> Annual International Pittsburgh Coal Conference (11-15 September 2000).

A modification was issued to the Repayment Agreement for the LPMEOH™ Demonstration Project. The term of the agreement and the method of calculating the amount of repayment were adjusted.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2000. Fifty-nine percent (59%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2000.

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## ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation (now ARCADIS Geraghty & Miller)
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU"
AFFTU	-	Alternative Fuels Field Trailer Unit
Balanced Gas	-	A syngas with a composition of hydrogen (H <sub>2</sub> ), carbon monoxide (CO), and carbon dioxide (CO <sub>2</sub> ) in stoichiometric balance for the production of methanol
Btu	-	British Thermal Unit
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Activity	-	the rate at which the catalyst promotes the desired chemical reaction to proceed within the limitations of chemical equilibrium
Catalyst Age ( $\eta$ -eta)	-	the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave)
Catalyst Concentration	-	Synonym for Slurry Concentration
Catalyst Loading	-	Synonym for Slurry Concentration
CO Conversion	-	the percentage of CO consumed across the reactor
Crude Grade Methanol	-	Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity; requires further distillation in existing Eastman equipment prior to use
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-NETL	-	The DOE's National Energy Technology Laboratory (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
FFV	-	flexible-fuel vehicle
Fresh Feed	-	sum of Balanced Gas, H <sub>2</sub> Gas, and CO Gas
Gas Holdup	-	the percentage of reactor volume up to the Gassed Slurry Height which is gas
Gassed Slurry Height	-	height of gassed slurry in the reactor
HAPs	-	Hazardous Air Pollutants
Hydrogen Gas	-	A syngas containing an excess of hydrogen (H <sub>2</sub> ) over the stoichiometric balance for the production of methanol; also called H <sub>2</sub> Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM	-	An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on
Inlet Superficial Velocity	-	the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second
K	-	Sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial gas facility at LaPorte, Texas, where the LPMEOH™ Process was successfully piloted
LPDME	-	Liquid Phase DME Process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
M85	-	a fuel blend of 85 volume percent methanol and 15 volume percent unleaded gasoline
MeOH	-	methanol
Methanol Productivity	-	the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)
MW	-	molecular weight, pound per pound mole

## ACRONYMS AND DEFINITIONS (cont'd)

NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
$\rho$	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	pounds per square inch
psia	-	pounds per square inch (absolute)
psig	-	pounds per square inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCF	-	Standard Cubic Feet
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	-	A gas containing primarily hydrogen (H <sub>2</sub> ) and carbon monoxide (CO), or mixtures of H <sub>2</sub> and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO <sub>2</sub> , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Unit and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
vol%	-	volume %
WBS	-	Work Breakdown Structure
wt	-	weight



## **Executive Summary**

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and is in operation at a site located at the Eastman chemicals-from-coal complex in Kingsport.

On 04 October 1994, Air Products and Eastman signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the project. These partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Modification No. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the project, and to act as the primary interface with DOE. As subcontractor to the Partnership, Air Products provided the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Unit, and is providing the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman is responsible for operation of the LPMEOH™ Process Demonstration Unit, and for the interconnection and supply of syngas, utilities, product storage, and other needed services.

The project involves the operation of an 80,000 gallons per day (260 tons per day (TPD)) methanol unit utilizing coal-derived syngas from Eastman's integrated coal gasification facility. The new equipment consists of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology to be demonstrated is the product of a cooperative development effort by Air Products and DOE in a program that started in 1981. Developed to enhance electric power generation using integrated gasification combined cycle (IGCC) technology, the LPMEOH™ Process is ideally suited for directly processing gases produced by modern day coal gasifiers. Originally tested at the Alternative Fuels Development Unit (AFDU), a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman chemicals-from-coal complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable

on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test program will be conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The operating test phase and off-site product-use test program will demonstrate the commercial viability of the LPMEOH™ Process and allow utilities to evaluate the application of this technology in the coproduction of methanol with electricity. A typical commercial-scale IGCC coproduction facility, for example, could be expected to generate 200 to 350 MW of electricity, and to also manufacture 45,000 to 300,000 gallons per day of methanol (150 to 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project has also been performing design verification testing (DVT), including laboratory- and pilot-scale research and market verification studies, to evaluate whether to include a demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Modification No. M009). After approval, the project initiated Phase 1 - Design - activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

The LPMEOH™ Demonstration Unit operated at 95% availability throughout the quarter. A 2-hour forced outage was caused by the failure of the control valve which regulates the flow of boiler feedwater to the steam drum on the LPMEOH™ Reactor. An additional 77-hour forced outage was initiated by a leak on instrument tubing associated with the measurement of boiler feedwater flow to the steam drum. A power transient was also experienced which interrupted the operation of the LPMEOH™ Demonstration Unit and adjoining process units within the chemicals-from-coal complex. In addition, a planned outage was taken on 13 June 2000 to conduct a test of the procedure for in-situ catalyst activation, and to perform an initial study of the ramping of the LPMEOH™ Reactor from a standby condition.

A major catalyst withdrawal and addition campaign was undertaken during April and May of 2000 to raise catalyst activity. A series of four withdrawals were conducted on 22 and 24 April 2000. This was followed by five catalyst additions which were activated and added between 25 April and 05 May 2000. After the addition of the fifth batch of catalyst, the total catalyst inventory was calculated to be 45,207 pounds.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average of 703 KSCFH. During these operating periods, the reactor pressure was set at between 700 and 710 psig and temperature was maintained at 235°C. Three intervals of carbon monoxide (CO)-rich testing were also performed.

On 21 March 2000, a syngas stream which contains primarily CO (CO Gas) was introduced with the Balanced Gas to achieve a ratio of hydrogen (H<sub>2</sub>) to CO in the reactor inlet of 1:1. Approximately 40 KSCFH of CO Gas was introduced with approximately 600 KSCFH of Balanced Gas for this CO-rich feed case. This case was concluded on 13 April 2000. Reactor pressure was adjusted to 695 psig during the period of CO Gas addition to allow for control of the makeup flow from the CO header.

On 09 May 2000, approximately 150 KSCFH of CO Gas was introduced with approximately 450 KSCFH of Balanced Gas to achieve a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.5:1. Reactor temperature was raised to 250°C during this test period to allow for greater catalyst productivity and to lower the purge rate. This case was concluded with the unit shutdown on 10 May 2000 after about 22 hours of operation.

On 22 June 2000, approximately 125 KSCFH of CO Gas was introduced with approximately 450 KSCFH of Balanced Gas to achieve a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.6:1. This case was concluded after about 6 hours of operation.

There were two extended periods of operation at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 1.24% per day was calculated for the period 24 March to 12 April 2000, during which the reactor inlet gas with a H<sub>2</sub>/CO ratio of 1:1 was fed to the LPMEOH™ Reactor. An overall deactivation rate of 0.89% per day was calculated for the period 13 May and 10 June 2000, during which Balanced Gas alone was used as feed to the reactor. The results of these two data sets are statistically similar, given the scatter in the calculated values for the catalyst rate constant. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples; however, the size increase has stabilized or decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in

December of 1997. The concentration of iron (another poison), although low (less than 200 ppmw), had been increasing, but has now stabilized in the most recent samples.

Eastman has accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon to replace the manganese oxide which is currently used in the 29C-40 catalyst guard bed within the LPMEOH™ Demonstration Unit. This material, along with other candidates which were recommended by vendors as having affinity for arsenic, was tested in the laboratory. The capacity of any of these adsorbents for arsenic could not be determined; this would require an on-line method to measure the presence of arsenic in the gas leaving the adsorbent test apparatus (no on-line analytical technique exists to measure arsenic in a syngas stream to the parts-per-billion concentration). The copper-impregnated activated carbon showed affinity for arsenic, and this class of adsorbent should also be able to remove sulfur and metal carbonyl species from the syngas. Plans call for a test of the effectiveness of this activated carbon on a slip stream of syngas at the LPMEOH™ Demonstration Unit during the week of 10 July 2000, and the changeout of the 29C-40 catalyst guard bed during the week of 31 July 2000.

A test of the conditions for the proposed in-situ activation of catalyst in the LPMEOH™ Reactor was performed. Nitrogen (N<sub>2</sub>) at 80 psig was introduced at the suction of the compressor, and the compressor developed a flowrate of 250 KSCFH and a discharge pressure of 102 psig. No operating problems were observed. The corresponding reactor inlet flowrate during the in-situ activation procedure is about 20% less than the basis which was used during confirmation of the procedure in the autoclave. These results will be evaluated to determine if additional laboratory or field testing will be required.

During the restart from the planned outage on 13 June 2000, a test of the ability to ramp the LPMEOH™ Reactor from a standby condition to production rates was performed. This test attempted to simulate the ramping that must be performed within the startup requirements of an IGCC facility. Other operating systems within the Eastman chemicals-from-coal complex, such as the impact of purge flow from the LPMEOH™ Demonstration Unit on the operation of the boiler system, were identified as areas which could limit the ability to test the ramping capabilities of the slurry reactor. As a result of excellent communication and teamwork by the operating staff at Eastman, these constraints were minimized during this test. A ramping rate of 3% of the design flow (990 KSCFH) per minute was achieved, which approaches the minimum target rate of 5% per minute. Based upon the experience gained from this test, it is expected that the goal of 5 to 10% change in design flow per minute, which meets the requirements for the IGCC system, can be met.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The performance to date has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 5,382,395 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 58.9 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl

acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on one of the project sites selected for the off-site, product-use test program. A draft of the paper entitled “Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine” was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000); the paper will be forwarded to DOE for review and comment. Testing continues on the impact of the trace mineral oil in the stabilized methanol on performance of the catalyst in the reformer test apparatus at the University of Florida. A sample of fresh mineral oil was sent to the University; this material will be blended with chemical-grade methanol for testing in the reformer, and will be used to evaluate potential designs for an oil filtration and removal system which could be incorporated into the flowsheet for a phosphoric acid fuel cell system.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report is under development which presents the results of the Fall 1999 design verification test at the LaPorte AFDU. A separate Topical Report on the market analysis for DME and review of the economics of the LPDME™ Process will be prepared following the release of the draft DVT Topical Report.

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary in order to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. Work was initiated to update the Demonstration Test Plan, given the change in the term of the Cooperative Agreement.

Two DOE quarterly review meeting were held during the reporting period. Major topics at the first meeting (10-11 April 2000 in Pittsburgh) included a review of the LPMEOH™ Demonstration Unit performance since the last meeting (January 2000), and an update on the status of the request for the no-cost time extension. At the second meeting (28 June 2000 at Air Products’ offices in Trexlertown, PA), additional discussion focused on the results of the N<sub>2</sub> flow test on the recycle compressor and the subsequent ramping study.

A draft of the paper entitled “Catalyst and Process Development for Liquid Phase DME Synthesis” was submitted to DOE for review. This paper will be presented at 17<sup>th</sup> Annual International Pittsburgh Coal Conference (11-15 September 2000).

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expended (as invoiced), as of 30 June 2000. Fifty-nine percent (59%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2000.

## **A. Introduction**

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership will own and operate the facility for the demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to “demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility.” The project has been demonstrating the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

The LPMEOH™ Process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

## **B. Project Description**

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 8,600 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H<sub>2</sub> Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- *Reaction Area* - Syngas preparation and methanol synthesis reaction equipment.
- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

- *Storage/Utility Area*

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

### **C. Process Description**

The LPMEOH™ Demonstration Unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream

feedstocks and in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

## **D. Results and Discussion**

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

### ***D.1 Off-Site Testing (Product-Use Demonstration)***

#### **Discussion**

The product-use test program, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, became outdated due in large part to changes within the power and chemical industries. This original product test program under-represented new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated product-use test program attempts for broader market applications and for commercial fuels comparisons. The objective of the product-use test program is to demonstrate commercial market applications for the “as produced” methanol as a replacement fuel and as a fuel supplement. Fuel economics will be evaluated for the “as produced” methanol for use in municipal, industrial, and utility applications and as fuel supplements for gasoline, diesel, and natural gas. These fuel evaluations will be based on the U.S. energy market needs projected during the 1998 to 2018 time period when the LPMEOH™ technology is expected to be commercialized.

The product-use test program has been developed to enhance the early commercial acceptance of central clean coal technology processing facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOH™ Process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt% water) which may be suitable for the premium fuel applications. When compared to conventional methanol synthesis processes, cost savings (10 to 15%) of several cents per gallon of methanol can be achieved in coproduction facilities, if the suitability of the stabilized product as a fuel can be demonstrated. The applications (for example, as a hydrogen source for fuel cells, and as a clean transportable, storable fuel for dispersed power) will require testing of the product to confirm its suitability. Chemical feedstock applications will also be tested as warranted.

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration unit is being made available for product-use tests. Product-use tests are targeted for an approximate 18 to 30-month period, and commenced during the first year of demonstration operations. An initial inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOH™ Demonstration Unit in February of 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory held at the LaPorte AFDU. Air Products,



ARCADIS Geraghty & Miller, and the DOE have worked together to select the projects to be included in the off-site, product-use test program.

Activity during this quarter

One of the product-use test projects has ongoing activities; Appendix B contains the status report for this work. The other six projects have completed testing of stabilized methanol, and are at stages of development of their respective final reports. Status and highlights include:

ARCADIS Geraghty & Miller Flexible-Fuel Vehicle (FFV) - The final report for this project was submitted to Air Products (no update in this reporting period).

Stationary Turbine for Nitrogen Oxide (NO<sub>x</sub>) Control - The test results on the low-NO<sub>x</sub> gas turbine combustor fueled with stabilized methanol from the LPMEOH™ Demonstration Unit have been prepared. Air Products is awaiting the submittal of the draft final report from ARCADIS Geraghty & Miller.

West Virginia University (WVU) Stationary Gas Turbine - Testing of stabilized methanol in the gas turbine system has been completed, and preparation of the final report is underway. A draft of the paper entitled “Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine” was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000); the paper will be forwarded to DOE for review and comment.

Aircraft Ground Equipment Emulsion - A final report on the use of a methanol emulsion as the fuel for a flight line generator at Tyndall Air Force Base was sent to Air Products; comments are pending.

University of Florida Fuel Cell - Testing continues on the impact of the trace mineral oil in the stabilized methanol on performance of the catalyst in the reformer test apparatus. In support of this diagnostic effort, a sample of fresh mineral oil was sent to the University; this material will be blended with chemical-grade methanol for testing in the reformer, and will be used to evaluate potential designs for an oil filtration and removal system.

West Virginia University Tri-Boro Bus - The draft final report for this project was submitted to Air Products (no update in this reporting period).

Florida Institute of Technology Bus & Light Vehicle - The final report on testing of stabilized methanol as a transportation fuel at the Florida Institute of Technology was received. DOE accepted Air Products’ recommendation to provide stabilized methanol from the LPMEOH™ Process Demonstration Unit for use as part a new contract between the Institute and the Florida Energy Office. Air Products will receive copies of the reports which are submitted to the State of Florida.

## ***D.2 DME Design Verification Testing***

The LPMEOH™ Demonstration Project has supported Design Verification Testing (DVT) to coproduce dimethyl ether (DME) with methanol via the Liquid Phase Dimethyl Ether (LPDME) Process. DVT was required to provide additional data for engineering design and evaluation of the potential for demonstration at the LPMEOH™ Demonstration Unit. The essential steps required for decision-making were: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

Initial market economic studies have shown that the LPDME Process should have a significant economic advantage for the coproduction of DME with methanol for local markets. The studies show that the market applications for DME are large. DME is an ultra clean diesel fuel. DME is a key intermediate in a commercial syngas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels. An LPDME catalyst system with reasonable long-term activity and stability was developed from the laboratory R&D work.

The LPDME Process is currently not applicable to hydrogen (H<sub>2</sub>)-rich syngas, and it was unlikely that a substantive LPDME demonstration would be recommended for Kingsport. Given these circumstances, the strategy for commercialization must combine the results of the following two areas:

- 1) catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the design verification testing at the LaPorte AFDU; and
- 2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot-plant scale.

Following this meeting, a formal recommendation to proceed with the interim campaign at the LaPorte AFDU was issued by Air Products to DOE. The objectives for this campaign, as stated in this recommendation, were:

- 1) Determine commercial viability of the LPDME Process on a 10 TPD scale, using commercially produced catalysts.
- 2) Obtain information to correlate scale-up of catalyst aging from the laboratory autoclave to the slurry bubble column.
- 3) Conduct process variable testing at conditions of potential commercial interest.

- 4) Perform experiments to better understand the hydrodynamics of the slurry bubble column.

DOE issued a letter dated 10 August 1999 accepting Air Products' recommendation to proceed with DME DVT activities at the LaPorte AFDU.

Execution of the LPDME design verification test at the LaPorte AFDU was completed during October and November of 1999, and preliminary results from the operation were presented in Technical Progress Report No. 22. Results from a cost estimate for a commercial-scale LPDME plant were presented in Technical Progress Report No. 23. After discussing the results from the LPDME Design Verification Testing activities and the ongoing performance results from Kingsport, the project participants agreed that the available resources should be directed toward improving the catalyst performance for the LPMEOH™ Process during the remaining time within the operating program; any improvement in the catalyst performance for the methanol synthesis catalyst will also yield benefits for the LPDME catalyst system.

A Topical Report is under development which presents the results of the design verification test at the LaPorte AFDU. A separate Topical Report on the market analysis for DME and review of the economics of the LPDME™ Process will be prepared by the LPMEOH™ Demonstration Project following the release of the draft DVT Topical Report.

### ***D.3 LPMEOH™ Process Demonstration Unit - Methanol Operation***

Table D.3-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix C contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

During the reporting period, a total of 5,382,395 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents or injuries were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 95% availability throughout the quarter. A 2-hour forced outage was caused by the failure of the control valve which regulates the flow of boiler feedwater to the steam drum on the LPMEOH™ Reactor. An additional 77-hour forced outage was initiated by a leak on instrument tubing associated with the measurement of boiler feedwater flow to the steam drum. A power transient was also experienced which interrupted the operation of the LPMEOH™ Demonstration Unit and adjoining process units within the chemicals-from-coal complex; maintenance was also performed on rupture disks in the cooling water system after the electrical trip. In addition, a planned outage was taken on 13 June 2000 to conduct a test of the procedure for in-situ catalyst activation, and to perform an initial study of the ramping of the LPMEOH™ Reactor from a standby

**Table D.3-1. Data Summary for LPMEOH™ Demonstration Unit**

Case	Date	Days	Gas	Temp	Pres.	Fresh	Recycle	Reactor	Purge	Inlet Sup.	Space	Slurry	Gas	Gassed	Catalyst	Catalyst	CO	Reactor		Syngas	Raw	Catalyst	Reactor	U		Sparger	Sparger
		On Stream				Feed (KSCFH)	Gas (KSCFH)	Feed (H <sub>2</sub> CO)	Gas (KSCFH)	Velocity (ft/sec)	Velocity (l/hr-kg)	Conc. (wt% ox)	Holdup (vol%)	Slurry Hgt (ft)	Inventory (lb)	Age (eta)	Conv. (%)	O-T-M Conv. (%)	Util. (SCF/lb)	MeOH Prod. (TPD)	MeOH Prod. (gmol/hr-kg)	Vol. Prod. (TPD/lb3)	Overall (Btu/hr ft2 F)	Sparger dP (psi)	Sparger Resist. (K")		
21	1-Apr-00	833	1:1	233	695	626	2,191	1.11	105.0	0.67	3740	42.1	31.8	54.0	44,687	0.346	15.8	18.4	43.6	183.0	10.70	0.081	108	7.00	4.33		
21	2-Apr-00	834	1:1	234	700	638	2,346	1.08	119.5	0.70	3940	42.1	22.4	47.5	44,687	0.327	14.6	17.3	44.8	181.8	10.64	0.091	130	7.61	4.25		
21	4-Apr-00	836	1:1	234	700	657	2,272	1.15	98.1	0.70	3934	46.6	31.0	45.0	44,687	0.325	15.2	17.7	43.0	183.4	10.71	0.097	122	7.62	4.36		
21	5-Apr-00	837	1:1	234	700	640	2,255	1.11	95.4	0.69	3867	43.0	32.2	52.5	44,687	0.327	14.9	17.7	42.4	180.8	10.50	0.082	117	7.18	4.18		
21	6-Apr-00	838	1:1	234	700	682	2,229	0.91	147.2	0.69	3849	42.7	31.5	52.5	44,687	0.312	12.5	16.5	46.8	174.9	10.26	0.079	113	7.24	4.29		
21	7-Apr-00	839	1:1	234	700	692	2,225	0.86	160.4	0.68	3843	42.5	27.3	50.0	44,687	0.312	12.1	16.3	48.0	173.2	10.14	0.082	126	7.38	4.28		
21	8-Apr-00	840	1:1	234	700	593	2,275	1.09	95.0	0.68	3842	45.3	26.6	44.5	44,687	0.305	13.9	16.6	43.5	163.8	9.57	0.088	135	7.90	4.49		
21	9-Apr-00	841	1:1	233	700	622	2,288	1.12	97.2	0.69	3885	43.9	31.8	50.5	44,687	0.306	14.1	16.7	43.2	172.7	10.10	0.081	120	7.08	4.17		
21	10-Apr-00	842	1:1	234	700	619	2,229	1.06	97.0	0.68	3802	44.1	33.0	51.0	44,687	0.327	14.3	17.2	42.8	173.2	10.13	0.081	131	6.90	4.09		
21	11-Apr-00	843	1:1	233	700	640	2,245	1.14	104.9	0.68	3841	44.7	32.6	49.5	44,687	0.308	14.5	16.9	44.5	172.7	10.10	0.083	106	6.80	4.10		
21	12-Apr-00	844	1:1	234	700	645	2,230	1.02	112.8	0.68	3813	44.7	34.7	51.0	44,687	0.316	13.7	17.0	45.0	172.0	10.06	0.080	99	6.98	4.12		
11	13-Apr-00	845	Balanced	233	700	648	2,173	2.77	100.5	0.66	3739	42.8	22.9	46.5	44,687	0.271	26.0	17.8	43.2	180.1	10.52	0.092	130	4.19	4.13		
11	14-Apr-00	846	Balanced	234	700	642	2,161	2.87	96.8	0.66	3697	43.0	29.6	50.5	44,687	0.266	26.5	17.8	42.9	179.6	10.50	0.085	122	4.20	4.37		
11	15-Apr-00	847	Balanced	234	700	655	2,181	2.81	109.8	0.66	3723	42.8	24.4	47.5	44,687	0.258	25.5	17.5	44.3	177.4	10.37	0.089	122	4.29	4.38		
11	21-Apr-00	853	Balanced	234	700	636	2,216	2.86	108.7	0.67	3750	43.6	28.8	49.0	44,687	0.244	24.9	16.9	44.5	171.5	10.02	0.083	124	4.53	4.56		
11	22-Apr-00	854	Balanced	235	700	631	2,196	3.00	110.5	0.66	3717	43.0	33.9	54.0	44,687	0.234	25.8	16.9	44.8	169.0	9.88	0.074	113	4.18	4.32		
11	24-Apr-00	856	Balanced	234	700	551	2,266	2.74	124.5	0.66	4851	38.0	23.7	43.5	34,207	0.247	19.1	13.6	47.9	137.9	10.54	0.076	139	4.28	4.40		
11	26-Apr-00	858	Balanced	234	700	550	2,236	4.48	74.3	0.64	4418	38.2	30.4	47.5	36,407	0.319	32.7	15.8	42.8	154.0	11.04	0.077	139	3.28	4.56		
11	27-Apr-00	859	Balanced	234	700	533	2,285	3.55	54.0	0.66	4555	40.7	33.2	47.5	36,407	0.291	27.2	15.6	41.1	155.5	11.15	0.078	132	3.97	4.40		
11	28-Apr-00	860	Balanced	234	700	614	2,292	2.77	89.4	0.68	4710	40.8	32.0	46.5	36,407	0.301	23.8	16.4	43.1	171.0	12.26	0.088	131	4.57	4.31		
11	29-Apr-00	861	Balanced	235	700	682	2,249	3.04	79.5	0.69	4465	40.3	28.0	47.5	38,607	0.345	29.4	19.0	41.7	196.1	13.26	0.098	125	4.65	4.40		
11	30-Apr-00	862	Balanced	234	700	706	2,162	3.65	73.1	0.67	4126	38.5	20.2	48.5	40,807	0.380	37.0	20.9	40.6	209.0	13.36	0.103	135	4.23	4.47		
11	1-May-00	863	Balanced	234	710	768	2,183	3.12	85.7	0.68	4245	38.8	21.0	48.5	40,807	0.395	34.6	21.9	41.1	224.3	14.35	0.110	135	4.74	4.36		
11	2-May-00	864	Balanced	234	710	776	2,182	3.50	88.9	0.68	4013	40.1	20.8	48.5	43,007	0.384	37.5	21.9	41.4	224.6	13.63	0.110	129	4.45	4.47		
11	3-May-00	865	Balanced	234	710	738	2,072	3.96	63.5	0.65	3830	40.3	18.2	46.5	43,007	0.411	42.6	22.4	40.3	218.7	13.27	0.112	149	3.97	4.52		
11	13-May-00	875	Balanced	234	709	784	2,130	3.43	69.0	0.67	3780	43.1	26.7	49.0	45,207	0.415	40.5	23.9	39.9	236.0	13.62	0.115	141	4.33	4.00		
11	14-May-00	876	Balanced	234	709	793	2,147	3.31	70.0	0.68	3833	45.5	29.6	46.5	45,207	0.405	38.8	23.5	40.2	236.8	13.67	0.121	142	4.43	3.97		
11	16-May-00	878	Balanced	233	708	880	2,094	3.16	138.7	0.68	3844	45.5	28.8	46.5	45,207	0.397	37.5	23.6	43.4	243.1	14.05	0.125	130	4.31	3.96		
11	17-May-00	879	Balanced	233	708	873	2,082	3.01	124.7	0.69	3864	41.2	20.4	48.5	45,207	0.397	26.2	23.6	43.0	243.6	14.08	0.120	119	4.49	3.95		
11	18-May-00	880	Balanced	234	708	854	2,091	3.07	109.2	0.69	3846	41.1	18.3	47.5	45,207	0.399	37.1	23.8	42.4	241.8	13.97	0.121	131	4.68	4.06		
11	19-May-00	881	Balanced	234	709	860	2,089	2.93	111.3	0.69	3850	43.8	27.2	48.0	45,207	0.397	36.0	23.9	42.4	243.7	14.08	0.121	128	4.70	4.00		
11	20-May-00	882	Balanced	233	710	638	2,195	3.88	27.7	0.66	3701	43.8	27.5	48.0	45,207	0.377	40.0	21.2	38.3	199.9	11.53	0.099	132	4.16	3.96		
11	25-May-00	887	Balanced	234	710	535	2,056	4.95	34.2	0.57	3204	42.9	23.0	47.0	45,207	0.390	41.9	18.7	39.6	162.1	9.35	0.082	138	2.62	4.61		
11	27-May-00	889	Balanced	234	710	795	2,087	3.61	102.9	0.66	3706	41.2	25.2	51.5	45,207	0.387	40.8	22.7	42.6	223.9	12.94	0.104	142	4.08	3.97		
11	28-May-00	890	Balanced	234	710	766	2,141	3.41	82.1	0.67	3792	44.2	25.9	46.5	45,207	0.360	37.3	21.7	41.0	223.8	12.92	0.115	151	4.3	4.22		
11	29-May-00	891	Balanced	234	710	765	2,159	3.41	87.6	0.67	3792	42.3	28.8	52.0	45,207	0.349	36.7	21.4	41.5	221.4	12.76	0.101	126	4.19	4.15		
11	30-May-00	892	Balanced	234	710	765	2,134	3.47	91.6	0.67	3752	41.6	22.4	49.0	45,207	0.347	37.1	21.4	41.7	220.0	12.70	0.107	131	3.96	4.11		
11	31-May-00	893	Balanced	234	710	765	2,096	3.46	92.2	0.66	3739	41.0	19.9	48.5	45,207	0.344	36.9	21.4	42.0	218.6	12.63	0.107	131	3.95	4.11		
11	1-Jun-00	894	Balanced	234	710	761	2,112	3.29	91.0	0.67	3758	40.9	23.4	51.0	45,207	0.346	35.7	21.4	41.5	219.8	12.70	0.103	123	4.07	4.06		
11	2-Jun-00	895	Balanced	233	710	764	2,116	3.17	93.5	0.67	3770	40.3	18.6	49.0	45,207	0.342	34.3	21.2	41.7	220.1	12.71	0.107	119	4.28	4.29		
11	3-Jun-00	896	Balanced	234	710	764	2,141	3.11	93.6	0.68	3807	41.0	18.8	48.0	45,207	0.337	33.5	21.1	41.7	219.6	12.68	0.109	122	4.33	4.20		
11	4-Jun-00	897	Balanced	234	710	748	2,120	3.21	96.9	0.67	3747	43.5	23.2	46.0	45,207	0.326	33.7	20.7	42.2	212.9	12.30	0.110	125	4.17	4.26		
11	5-Jun-00	898	Balanced	234	710	746	2,122	3.21	81.1	0.67	3752	44.0	25.2	46.5	45,207	0.333	34.5	21.2	41.2	217.2	12.55	0.111	138	4.29	4.34		
11	6-Jun-00	899	Balanced	234	710	741	2,180	2.93	65.2	0.68	3842	45.6	26.4	44.5	45,207	0.336	32.4	21.2	40.1	221.7	12.79	0.119	146	4.71	4.33		
11	8-Jun-00	901	Balanced	234	710	759	2,142	3.02	94.2	0.68	3800	43.7	24.6	46.5	45,207	0.329	32.4	21.0	41.8	218.0	12.59	0.112	127	4.39	4.27		
11	9-Jun-00	902	Balanced	234	710	759	2,126	2.79	87.0	0.67	3775	45.6	25.6	44.0	45,207	0.332	31.4	21.3	41.4	220.0	12.70	0.119	138	4.63	4.33		
11	10-Jun-00	903	Balanced	234	710	765	2,104	2.76	93.0	0.67	3763	45.0	24.0	44.0	45,207	0.329	30.9	21.2	42.1	218.1	12.60	0.118	135	4.54	4.26		
11	14-Jun-00	907	Balanced	235	710	744	2,137	2.92	79.3	0.67	3760	43.4	26.0	48.0	45,207	0.320	32.0	20.9	41.3	216.1	12.48	0.107	120	4.86	4.68		
11	15-Jun-00	908	Balanced	235	710	753	2,132	3.00	88.1	0.67	3783	43.3	25.8	48.0	45,207	0.320	32.4	20.9	41.7	216.7	12.52	0.108	122	4.73	4.56		
11	16-Jun-00	909	Balanced	236																							

condition. Appendix D, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

#### Catalyst Life (eta) – March - June 2000

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable eta ( $\eta$ ), which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix D, Figure 1 plots  $\log \eta$  versus days onstream from September of 1999 to the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of  $\log \eta$  is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

A major catalyst withdrawal and addition campaign was undertaken during April and May of 2000 to raise catalyst activity. A series of four withdrawals were conducted on 22 and 24 April 2000. This was followed by five catalyst additions which were activated and added between 25 April and 05 May 2000. After the addition of the fifth batch of catalyst, the total catalyst inventory was calculated to be 45,207 pounds.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 703 KSCFH. During these operating periods, the reactor pressure was set at between 700 and 710 psig and temperature was maintained at 235°C. Three intervals of CO-rich testing were also performed, and are documented below.

On 21 March 2000, CO Gas was introduced with the Balanced Gas to achieve a reactor inlet feed H<sub>2</sub>/CO ratio of 1:1. Approximately 40 KSCFH of CO Gas was introduced with approximately 600 KSCFH of Balanced Gas for this CO-rich feed case. This case was concluded on 13 April 2000. Reactor pressure was adjusted to 695 psig during the period of CO Gas addition to allow for control of the makeup flow from the CO header.

On 09 May 2000, a large amount of CO Gas (up to 200 KSCFH) became available to be introduced with the Balanced Gas to achieve a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.5:1. Approximately 150 KSCFH of CO Gas was introduced with approximately 450 KSCFH of Balanced Gas for this CO-rich feed case. Reactor temperature was raised to 250°C during this test period to allow for greater catalyst productivity and to lower the purge rate. Limitations in the maximum purge rate that could be handled by the downstream boiler system resulted in less than the maximum CO Gas rate being processed. This case was concluded with the unit shutdown on 10 May 2000 after about 22 hours of operation.

On 22 June 2000, a large quantity of CO Gas (up to 200 KSCFH) also became available to be introduced with the Balanced Gas to achieve a reactor inlet feed H<sub>2</sub>/CO ratio of about 0.6:1. Approximately 125 KSCFH of CO Gas was introduced with approximately 450 KSCFH of Balanced Gas for this CO-rich feed case. This case was concluded after about 6 hours of operation.

There were two extended periods of operation (minimum 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 1.24% per day was calculated for the period 24 March to 12 April 2000, during which the reactor inlet gas with a H<sub>2</sub>/CO ratio of 1:1 was fed to the LPMEOH™ Reactor. An overall deactivation rate of 0.89% per day was calculated for the period 13 May and 10 June 2000, during which Balanced Gas alone was used as feed to the reactor. The results of these two data sets are statistically similar, given the scatter in the calculated values for the catalyst rate constant. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix D, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic (to around 1,200 ppmw), which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to increase (present levels are at about 300 ppmw). Copper crystallite size measurements have shown an increase in the most recent samples; however, the size increase has stabilized or decreased over the last few samples. Other methods of crystallite size determination have corroborated the increased size measurements. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison), although low (less than 200 ppmw), had been increasing, but has now stabilized in the most recent samples.

Air Products has been performing laboratory screening experiments to identify whether different adsorbents are available which can remove trace levels of arsenic and sulfur from syngas within the catalyst guard bed system at the LPMEOH™ Demonstration Unit. Eastman has accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon to replace the manganese oxide which is currently used in the 29C-40 catalyst guard bed within the LPMEOH™ Demonstration Unit. This material, along with other candidates which were recommended by vendors as having affinity for arsenic, was tested in the laboratory. The capacity of any of these adsorbents for arsenic could not be determined; this would require an on-line method to measure the presence of arsenic in the gas leaving the adsorbent test apparatus (no on-line analytical technique exists to measure arsenic in a syngas stream to the parts-per-billion concentration). The copper-impregnated activated carbon showed affinity for arsenic, and this class of adsorbent should also be able to remove sulfur and metal carbonyl species from the syngas. Plans call for a test of the effectiveness of this activated carbon on a slip stream of syngas at the LPMEOH™ Demonstration Unit during the week of 10 July 2000, and the changeout of the 29C-40 catalyst guard bed during the week of 31 July 2000.

#### In-situ Catalyst Activation

A test of the conditions for the proposed in-situ activation of catalyst in the LPMEOH™ Reactor was performed. This involved a flow test of the recycle compressor on low pressure N<sub>2</sub> in order to verify the operating pressure and flowrate which can be developed. N<sub>2</sub> at 80

psig was introduced at the suction of the compressor, and the compressor developed a flowrate of 250 KSCFH and a discharge pressure of 102 psig. No operating problems were observed. The corresponding reactor inlet flowrate during the in-situ activation procedure is about 20% less than the basis which was used during confirmation of the procedure in the autoclave. These results will be evaluated to determine if additional laboratory or field testing will be required.

#### Initial Ramping Study

During the restart from the planned outage on 13 June 2000, a test of the ability to ramp the LPMEOH™ Reactor from a standby condition to production rates was performed. This test attempted to simulate the ramping that must be performed within the startup requirements of an IGCC facility. Other operating systems within the Eastman chemicals-from-coal complex, in particular the limits on rate-of-change on the parallel fixed-bed methanol plant and the impact of purge flow from the LPMEOH™ Demonstration Unit on the operation of the boiler system, were identified as areas which could limit the ability to test the ramping capabilities of the slurry reactor. As a result of excellent communication and teamwork by the operating staff at Eastman, these constraints were minimized during this test. A ramping rate of 3% of the design flow (990 KSCFH) per minute was achieved, which approaches the minimum target rate of 5% per minute. Based upon the experience gained from this test, it is expected that the goal of 5 to 10% change in design flow per minute, which meets the requirements for the IGCC system, can be met.

#### Sparger Resistance

The performance of the new sparger continues to exceed the design expectations for pressure drop and reactor operation. Appendix D, Figure 2 plots the average daily sparger resistance coefficient for the period following the March 1999 outage. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1.

### ***D.4 Planning and Administration***

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary in order to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. Work was initiated to update the Demonstration Test Plan, given the change in the term of the Cooperative Agreement.

Two DOE quarterly review meeting were held during the reporting period. Major topics at the first meeting (10-11 April 2000 in Pittsburgh) included a review of the LPMEOH™ Demonstration Unit performance since the last meeting (January 2000), and an update on the status of the request for the no-cost time extension. At the second meeting (28 June 2000 at Air Products' offices in Trexlertown, PA), additional discussion focused on the results of the N<sub>2</sub> flow test on the recycle compressor and the subsequent ramping study. The agenda,

extracts from the handouts, and the notes for each meeting are included in Appendix E and F.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 2000, are included in Appendix G. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2000. Fifty-nine percent (59%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2000.

The monthly reports for April, May, and June were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

A draft of the paper entitled “Catalyst and Process Development for Liquid Phase DME Synthesis” was submitted to DOE for review. This paper will be presented at 17<sup>th</sup> Annual International Pittsburgh Coal Conference (11-15 September 2000).

A modification was issued to the Repayment Agreement for the LPMEOH™ Demonstration Project. The term of the agreement and the method of calculating the amount of repayment were adjusted.

#### **E. Planned Activities for the Next Quarter**

- Continue to analyze catalyst slurry samples and reactor performance data to determine causes for deactivation of methanol synthesis catalyst.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on increasing catalyst activity, monitoring the performance of the gas sparger in the reactor, and finalizing the procedure for in-situ catalyst activation.
- Complete the changeout of the adsorbent materials in the 29C-40 catalyst guard bed within the LPMEOH™ Demonstration Unit to increase the removal efficiency of arsine and sulfur.
- Issue the draft Topical Report on the Fall 1999 LPDME design verification test at the LaPorte AFDDU.
- Submit an update to the Demonstration Test Plan to DOE for review and comment.
- Continue execution of the Off-Site, Product-Use Test Program.
- Conduct a Project Review Meeting with DOE.



## **F. Conclusion**

The LPMEOH™ Demonstration Unit operated at 95% availability throughout the quarter. A 2-hour forced outage was caused by the failure of the control valve which regulates the flow of boiler feedwater to the steam drum on the LPMEOH™ Reactor. An additional 77-hour forced outage was initiated by a leak on instrument tubing associated with the measurement of boiler feedwater flow to the steam drum. A power transient was also experienced which interrupted the operation of the LPMEOH™ Demonstration Unit and adjoining process units within the chemicals-from-coal complex. In addition, a planned outage was taken on 13 June 2000 to conduct a test of the procedure for in-situ catalyst activation, and to perform an initial study of the ramping of the LPMEOH™ Reactor from a standby condition.

A major catalyst withdrawal and addition campaign was undertaken during April and May of 2000 to raise catalyst activity. A series of four withdrawals were conducted on 22 and 24 April 2000. This was followed by five catalyst additions which were activated and added between 25 April and 05 May 2000. After the addition of the fifth batch of catalyst, the total catalyst inventory was calculated to be 45,207 pounds.

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the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples; however, the size increase has stabilized or decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison), although low (less than 200 ppmw), had been increasing, but has now stabilized in the most recent samples.

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A test of the conditions for the proposed in-situ activation of catalyst in the LPMEOH™ Reactor was performed. N<sub>2</sub> at 80 psig was introduced at the suction of the compressor, and the compressor developed a flowrate of 250 KSCFH and a discharge pressure of 102 psig. No operating problems were observed. The corresponding reactor inlet flowrate during the in-situ activation procedure is about 20% less than the basis which was used during confirmation of the procedure in the autoclave. These results will be evaluated to determine if additional laboratory or field testing will be required.

During the restart from the planned outage on 13 June 2000, a test of the ability to ramp the LPMEOH™ Reactor from a standby condition to production rates was performed. This test attempted to simulate the ramping that must be performed within the startup requirements of an IGCC facility. Other operating systems within the Eastman chemicals-from-coal complex, such as the impact of purge flow from the LPMEOH™ Demonstration Unit on the operation of the boiler system, were identified as areas which could limit the ability to test the ramping capabilities of the slurry reactor. As a result of excellent communication and teamwork by the operating staff at Eastman, these constraints were minimized during this test. A ramping rate of 3% of the design flow (990 KSCFH) per minute was achieved, which approaches the minimum target rate of 5% per minute. Based upon the experience

gained from this test, it is expected that the goal of 5 to 10% change in design flow per minute, which meets the requirements for the IGCC system, can be met.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The performance to date has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 5,382,395 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 58.9 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on one of the project sites selected for the off-site, product-use test program. A draft of the paper entitled “Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine” was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000); the paper will be forwarded to DOE for review and comment. Testing continues on the impact of the trace mineral oil in the stabilized methanol on performance of the catalyst in the reformer test apparatus at the University of Florida. A sample of fresh mineral oil was sent to the University; this material will be blended with chemical-grade methanol for testing in the reformer, and will be used to evaluate potential designs for an oil filtration and removal system which could be incorporated into the flowsheet for a phosphoric acid fuel cell system.

Activities associated with Design Verification Testing of the LPDME Process have been completed. A Topical Report is under development which presents the results of the Fall 1999 design verification test at the LaPorte AFDU. A separate Topical Report on the market analysis for DME and review of the economics of the LPDME™ Process will be prepared following the release of the draft DVT Topical Report.

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary in order to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. Work was initiated to update the Demonstration Test Plan, given the change in the term of the Cooperative Agreement.

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A modification was issued to the Repayment Agreement for the LPMEOH<sup>TM</sup> Demonstration Project. The term of the agreement and the method of calculating the amount of repayment were adjusted.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH<sup>TM</sup> Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2000. Fifty-nine percent (59%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2000.

## **APPENDICES**

### **APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM**

## **APPENDIX B - OFF-SITE TESTING (DEFINITION AND DESIGN)**

### **Quarterly Report:**

**University of Florida Fuel Cell (six pages)**

## **APPENDIX C - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS**

## **APPENDIX D - RESULTS OF DEMONSTRATION UNIT OPERATION**

**Table 1 - Summary of LPMEOH<sup>TM</sup> Demonstration Unit Outages -  
April/June 2000**

**Table 2 - Summary of Catalyst Samples - Second Catalyst Batch**

**Figure 1 - Catalyst Age ( $\eta$ ): September 1999 – June 2000**

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream**



**Table 1**  
**Summary of LPMEOH™ Demonstration Unit Outages - April/June 2000**

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
4/1/00 00:00	4/1/00 07:30	7.5	2.0	FV-106 Failure
4/1/00 09:30	4/16/00 17:00	367.5	102.5	FT-106 Leak
4/20/00 23:30	4/25/00 08:13	104.7	2.6	Syngas Outage
4/25/00 10:50	5/10/00 14:30	363.7	42.3	Electrical Trip, Rupture Disks
5/12/00 08:49	6/13/00 01:00	760.2	11.7	Planned Outage - Tests
6/13/00 12:41	6/30/00 23:59	419.3		End of Reporting Period
Total Operating Hours			2022.9	
Total Elapsed Hours			2184.0	
<b>Plant Availability, %</b>			<b>95.09</b>	

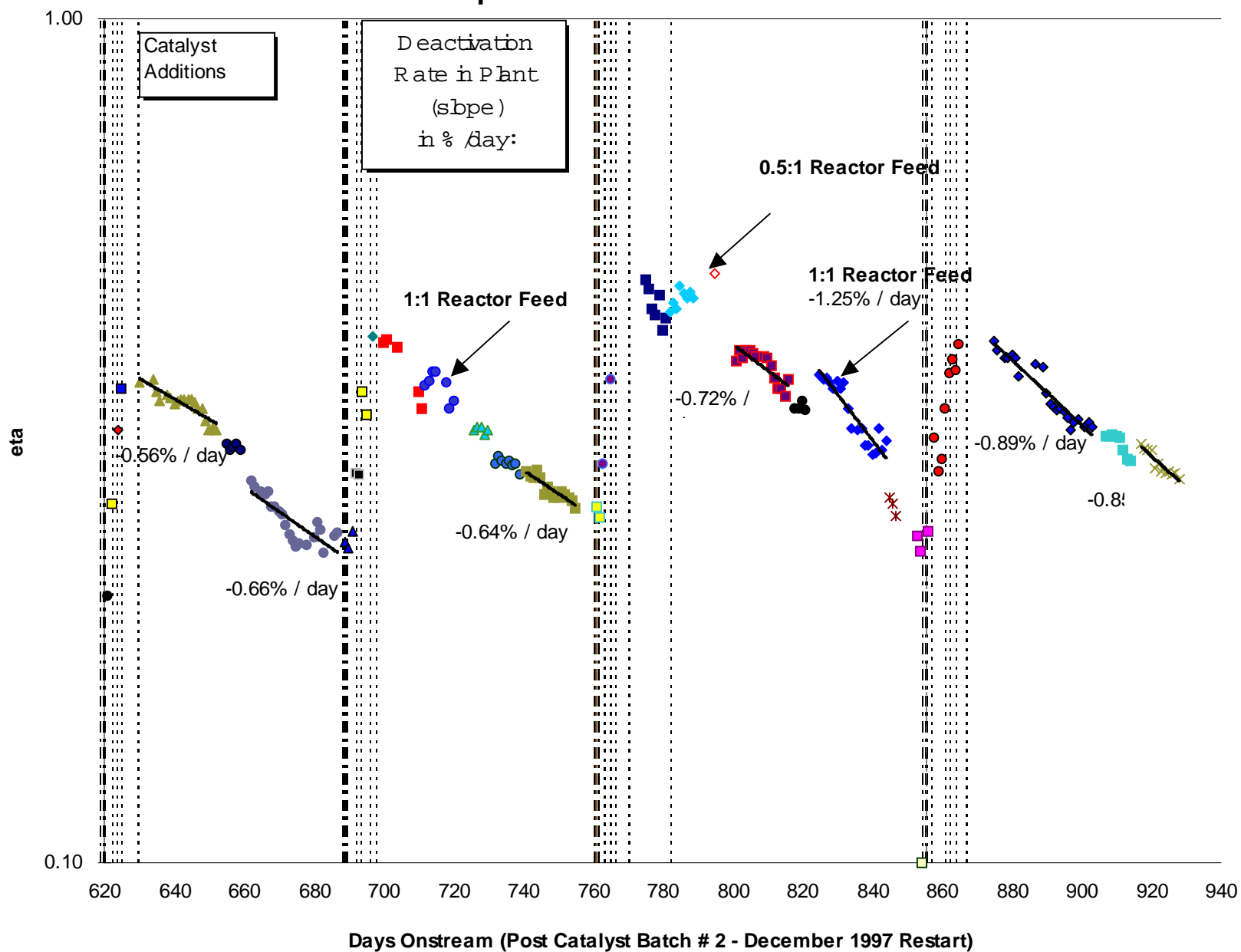
**Table 2**  
**Summary of Catalyst Samples - Second Catalyst Batch**

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m <sup>2</sup> /g	Fe	Ni	S	As	Cl
K9804-1	Reduction Sample 4/2/98 - Alternative Catalyst	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample from 29D-02 to Reactor	95.3	74		362	47.2	66.7	10.2	nd
K9712-2	Reactor Sample Day 1	100	123.8	75	92.1	<=18	<=167	<50	nd
K9712-3	Reactor Sample Day 4	130.9	64						
K9712-4	Reactor Sample Day 10	126.8	73.3	73	126	<=22	<=127	<50	nd
K9801-2	Reactor Sample 1/26/98	132.05	98.3		63.5	39.5	42.7	29.2	<100
K9802-1	Reactor Sample 2/3/98	141.1	91.5						
K9802-2	Reactor Sample 2/9/98	158.1	113						
K9802-3	Reactor Sample 2/15/98	145.7	91		67.1	36	<=97	209	
K9802-4	Reactor Sample 2/23/98	176.8	114.5						
K9803-2	Reactor Sample 3/10/1998	154.3	95.8	44	61.4	35.8	<=94	408	
K9803-4	Reactor Sample 3/29/98	169.6	87.9						
K9804-2	Reactor Sample 4/14/98	152.4	89.3		81.7	30.8	<=170	615	
K9805-2	Reactor Sample 5/11/98	219.2	109.6		73.15	35.85	163	538	
K9606-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5	61.4						
K9902-1	Reactor Sample 2/17/99	474.7	133.6		82.6	22.2	385	1490	<300
K9904-3	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
K9906-1	Reactor Sample 6/1/99	517	105	43	109	19.7	316	1680	40
K9907-1	Reactor Sample 7/13/99	446	116	59	175	19.7	488	1810	30
K9908-2	Reactor Sample 8/31/99	632	117	56	161	15.1	406	1470	50
K9909-2	Reactor Sample 9/21/99	357	109	64	132	11.2	253	1050	nd
K9910-2	Reactor Sample 10/19/99	135	94	55	157	15.4	343	1270	30
K9911-1	Reactor Sample 11/4/99				184	12.8	335	1580	na
K9912-1	Reactor Sample 12/8/99	797	121	60	167	13.9	248	1400	40
K0001-1	Reactor Sample 1/5/00	613	105	63	199	10.8	292	1190	nd
	Reactor Sample 1/19/00				205	10.0	432	1250	na
	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20

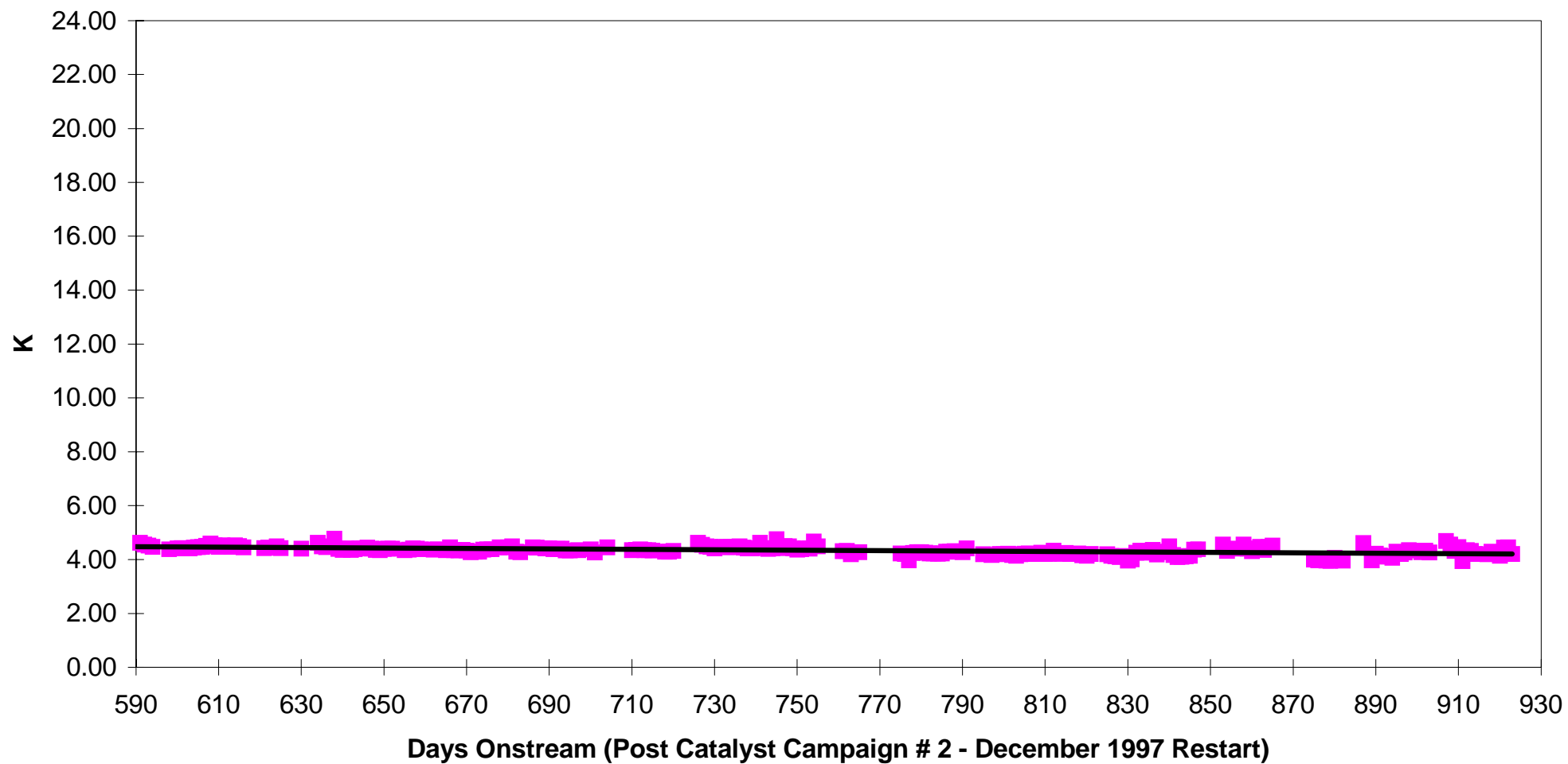
Notes:

- 1) nd = none detected
- 2) \* - these values represent re-analysis of the sample as compared to Technical Progress Report no. 17
- 3) na = data not available

**Figure 1 - Kingsport LPMEOH™ Catalyst Age (eta):  
September 1999 - June 2000**



**Figure 2 - Kingsport LPMEOH™  
Sparger Resistance Coefficient**



## **APPENDIX E - PROJECT REVIEW MEETING (10-11 APRIL 2000)**

## **APPENDIX F - PROJECT REVIEW MEETING (28 JUNE 2000)**

**APPENDIX G - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT  
REPORTS**